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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: WEST

Application No.: 10/601,234 Examiner: Stadler, Rebecca M.

Date Filed: June 20, 2003 Group: 1754

For: **METHOD FOR CONTINUOUS PRODUCTION OF A HYDRATE COMPOSITE**

CERTIFICATE OF TRANSMISSION/MAILING

I hereby certify that this correspondence is being facsimile transmitted to the USPTO or deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: MS RCE, Commissioner for Patents, Alexandria, VA 22313-1450 on 5/30/06

Robert J. Lauf Reg. No. 54,252

Robert J. Lauf

DECLARATION UNDER 37 C.F.R. §1.132

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I, Roddie R. Judkins, declare as follows:

1. I hold a BS in Engineering Chemistry and an MS in Chemistry from Tennessee Technological University, and a PhD in Physical Chemistry from Georgia Institute of Technology as described in more detail in my cv appended hereto.

2. For the past 28 years I have worked regularly in the field of engineering materials and on broader issues relating to fossil energy technology. I am quite familiar with the work of Dr. West and her team regarding the production of novel forms of gas

hydrates. I am currently employed as the Fossil Energy Program Director at Oak Ridge National Laboratory.

3. I have substantial expertise in the field of gas hydrate technology and chemistry and have authored or co-authored numerous publications, some of which are listed on my c.v. In addition, I am founder of the Interlaboratory Working Group on Methane Hydrates, which places me in a unique position to evaluate the novelty of projects in the context of the general state of the art.

4. I have reviewed the Final Office Action dated April 14, 2006 and the Examiner's assertions therein, as well as the cited references.

5. I have reviewed Application No. 10/601,234 entitled "METHOD FOR CONTINUOUS PRODUCTION OF A HYDRATE COMPOSITE" and the data provided therewith. I have copied independent claim 1 below, as now presented by Applicants current amendment (shown in italics).

1. A method for continuous production of a hydrate-containing material comprising the steps of:

flowing a hydrate-forming fluid through a pressurized, temperature-controlled, continuous-flow reactor as a continuously flowing fluid;

injecting water into said continuously-flowing hydrate-forming fluid, said water injected as a dispersed fluid at a Reynolds number characterizing the turbulent spraying regime to produce an emulsion of the two fluids; and,

allowing said emulsion to flow through said continuous-flow reactor for a sufficient time to allow solid gas hydrate particles to form while providing sufficient lateral restraint to consolidate said particles into a substantially solid extruded body.

6. The independent claim, as now presented, clearly points out a major element of novelty in Applicants' process, namely that the product is a **monolithic extrusion** rather than **individual hydrate granules**. Although the word "extrusion" does not appear in the specification as filed, the term is clearly supported by consideration of the drawings as filed. FIG. 1 clearly illustrates a lower spatial density of the individual hydrate particles when they are first forming, and a progressively higher packing density as the mixture travels along the tube, ultimately being discharged as a "consolidated mass". The tube wall clearly provides lateral restraint during this process. In my opinion, the term "substantially solid extruded body" is a clear and accurate description of the product as shown in FIG. 1 and therefore does not represent new matter. Furthermore, FIGS. 2 and 3 present actual photographs of the process, where, again, it is clear that the process is fundamentally an extrusion process.

Similar arguments will apply to claim 11 as currently amended by Applicants. The two independent claims have a clear relationship to each other, namely, the

recognition that one can make an emulsion of two fluids by spraying either fluid into the other.

7. According to the Examiner regarding claims 1-11:

Finally, claim 1 requires that a "consolidated solid-like hydrate/fluid/water" stream is formed. Iijima does not use the same language to describe the product that it delivers to the bottom of the ocean. However, the product of Iijima has negative buoyancy, as evinced by its description of the hydrate as something that will sink to the bottom of the ocean (see abstract, lines 5-6). The product of the present invention also possess negative buoyancy (see specification page 10, lines 1-2). This demonstrates that the product of Iijima meets the limitation of a "consolidated solid-like hydrate/fluid/water" stream being formed. Further, the process of Iijima in view of Max is the same as that claimed in the present invention. Therefore, the Iijima in view of Max process will form the same "consolidated solid-like hydrate/fluid/water" stream product.

Finally, claim 1 requires that a "consolidated solid-like hydrate/fluid/water" stream is formed. Spencer does not use the same language to describe the product that it delivers to the bottom of the ocean. However, the product of Spencer has negative buoyancy, as evinced by its description of the hydrate as something that will sink to the bottom of the ocean (see column 2, lines 6-13). The product of the present invention also possess negative buoyancy (see specification page 10, lines 1-2). This demonstrates that the product of Spencer meets the limitation of a "consolidated solid-like hydrate/fluid/water" stream being formed. Further, the process of Spencer in view of Max is the same as that claimed in the present invention. Therefore, the Spencer in view of Max process will form the same "consolidated solid-like hydrate/fluid/water" stream product.

As to applicant's argument that Iijima produces "only less-consolidated CO_2-hydrate-water forms," no standard of consolidation is taught in the present specification. Further, there is no basis in the art to distinguish one hydrate from another on the basis of "consolidation".

Regarding claim rejections under 35 USC § 103 based on cited art:

The process of Spencer is the same as that of the present invention, with the exception that Spencer does not disclose turbulent flow. Spencer is directed to a process for forming a hydrate to be sunk in the ocean. As such, it would be desirable desires to produce as much hydrate as possible in the Spencer process. In light of the teaching of Max, turbulent flow conditions in the Spencer process would ensure adequate hydrate formation. Therefore, it would be obvious to run the Spencer process under turbulent conditions, which would then be the same process as that claimed and as such the "consolidated solid-like hydrate/fluid/water stream" would be formed.

As to claim 7, Spencer '891 does not disclose a jet pump to control the water flow. However, Allen '137 does disclose the use of a jet pump in a similar method. It would have been obvious to one of ordinary skill in the art at the time of this invention to use the jet pump of Allen '137 in place of the regular pump of Spencer '891 in order to provide for additional mixing.

As to claim 5, Spencer '891 does not disclose the use of a mass flow controller to control the flow of carbon dioxide. Satek '886 does use a mass flow controller to control the flow of the feed mixture (see column 15, lines 1-2). It would have been obvious to one of ordinary skill in the art at the time of this invention to add the mass flow controller of Satek '866 to the Spencer in view of Max method in order to more precisely control the flow rate to the process for better overall quality.

As to claims 3 and 8, Spencer '891 does provide for adequate mixing of the carbon dioxide and the water, although the reference does not disclose the use of baffles. However, Ohsol '762 does use baffles for mixing in its process (see column 4, lines 39-43). It would have been obvious to one of ordinary skill in the art at the time of this invention to use static mixer blades in order to adequately mix the carbon dioxide and the water, while minimizing system complexity.

The process of Iijima is the same as that of the present invention, with the exception that Iijima does not disclose turbulent flow. Iijima is directed to a process for forming a hydrate to be sunk in the ocean. As such, it would be desirable to produce as much hydrate as possible in the Iijima process. In light of the teaching of Max, turbulent flow conditions in the Iijima process would ensure adequate hydrate formation. Therefore, it would be obvious to run the Iijiima process under turbulent conditions, which would then be the same process as that claimed and as such the "consolidated solid-like hydrate/fluid/water stream" would be formed.

As to claim 7, Iijima '611 does not disclose a jet pump to control the water flow. However, Allen '137 does disclose the use of a jet pump in a similar method. It would have been obvious to one of ordinary skill in the art at the time of this invention to use the jet pump of Allen '137 in place of the regular pump of Iijima '611 in order to provide for additional mixing. As discussed in Allen '137, a jet pump "contributes to the mixing of water with the mixture because of the high energy at which the jet pump injects water into the mixture (see col. 14, lines 50-53).

As to claim 5, Iijima '611 does not disclose the use of a mass flow controller to control the flow of carbon dioxide. Satek '886 does use a mass flow controller to control the flow of the feed mixture (see column 15, lines 1-2). It would have been obvious to one of ordinary skill in the art at the time of this invention to add the mass flow controller of Satek '866 to the Iijima in view of Max method in order to more precisely control the flow rate to the process for better overall quality.

As to claims 3 and 8, Iijima '611 does provide for adequate mixing of the carbon dioxide and the water, although the reference does not disclose the use of baffles. However, Ohsol '762 does use baffles for mixing in its process (see column 4, lines 39-43). It would have been obvious to one of ordinary skill in the art at the time of this invention to use baffles in order to adequately mix the carbon dioxide and the water, while minimizing system complexity.

8. I have been advised by Applicants' counsel that the issues raised by the Examiner relate to (i) whether the hydrate material disclosed in the present application as filed and the "substantially solid-like material" described in the cited art are the same

material, i.e., whether there is a basis in the art to distinguish one hydrate from another on the basis of "consolidation"; and (ii) whether Applicants' process is "obvious" based on consideration of the cited references taken alone or in combination.

9. Regarding (i), whether the material of the present application is the same as material disclosed in the cited art: the Examiner asserts that "the product of Iijima has negative buoyancy, as evinced by its description of the product as something that will sink to the bottom of the ocean... The product of the present invention also possess [sic] negative buoyancy... This demonstrates that the product of Iijima meets the limitation of a 'consolidated solid-like hydrate/fluid/water' stream being formed."

I note first of all that buoyancy *per se* does not prove whether or not a particular product is solid. Many liquids are known that are immiscible in water and have greater density than that of seawater; droplets of such liquids would therefore have negative buoyancy and would sink in the ocean. Ice, on the other hand, is solid and yet has positive buoyancy.

Granted, however, that the products of some of the cited art are, strictly speaking, solid particles or granules, the key difference lies in the issue of consolidation. The issues are best understood by analogy: individual snowflakes and snowballs are both, in a fundamental sense, "solids" and both are denser than air. At the same time, however, they are palpably different materials in many obvious ways. The individual snowflake has many times greater ratio of surface area to mass; it will melt immediately

if placed into water that is above its freezing point. The snowball, by contrast, will remain solid much longer even under conditions where it would not be stable indefinitely. Such greater persistence under metastable conditions gives Applicants' extruded mass a greater leeway to survive and sink in the ocean than the granular particles made by the cited art. The cited art processes discharge a "stream" or slurry of individual solid particles freely dispersed in seawater and do not yield a consolidated extrusion able to retain integrity as a solid body as Applicants' process does.

10. Regarding (ii), whether Applicants' process is "obvious" based on consideration of the cited references taken alone or in combination: I now bring attention to the following subject matter disclosed in Application No. 10/601,234:

In the photographs shown in FIGS. 2 and 3, a substantially monolithic extruded product is clearly being formed. It clearly has negative buoyancy as indicated by the fact that a length of the extruded material can be seen bending as the free length sags under the force of gravity. At the same time, it clearly has sufficient mechanical integrity to withstand significant deformation. By contrast, the teachings of the cited art are clearly directed to the production of substantially granular or particulate hydrate forms:

Iijima et al. '611 discloses several modes of making gas hydrates, all of which produce a substantially granular or particulate product. FIG. 7 shows solid hydrate being formed on the walls of a pipe, with a rotating screw blade to scrape the solid material off the walls as described at Col. 10 lines 49-55. FIG. 8 shows an arrangement

that does not have the rotating screw but is still shown as discharging isolated particles or flakes. Likewise, FIGS. 10 and 11 show other configurations in which the solid hydrate phase is depicted as isolated particles rather than a compacted body. The discussion of FIG. 11 at Col. 12 lines 15-18 specifically refers to "dispersing the product carbon dioxide hydrate out of the device". **Dispersing** particles is essentially the opposite of Applicants' **consolidation** process.

Max '034 contemplates the formation of individual hydrate particulates (referred to as "crystals" at Col. 2 line 20 and elsewhere). Because Max intends for the hydrate to form in one stage of the process and decompose in another stage of the process, consolidating the hydrate into a monolithic extruded body would work against Max's goals. Max also refers to the "hydrate slurry" (Col. 8 line 31 and elsewhere) which clearly refers to a suspension of individual particles in a fluid; this is also shown in FIG. 8 of Max where the hydrate phase is shown as individual particles that are freely dispersed in the flowing water and are not compacted into a monolithic body. The slurry of Max would clearly not have the mechanical properties shown by Applicants' material in Applicants' FIG. 3 as filed.

Spencer et al. '891 discuss the formation of hydrates that are "removed from the hydrate production chamber by conduit..." (Col. 6 lines 41-42) and specifically describe an operation in which the hydrates will be "released as solids into the ocean ... or pumped as a 50:50 clathrate-water slurry..." This clearly implies the formation of dispersed solid particles rather than a compacted monolithic body.

Ohsol '762 teaches the use of mixing baffles in a completely different process. Satek '886 teaches the use of a mass flow controller in a completely different process. Allen '137 teaches the use of a jet pump for mixing fluids. Because neither Spencer '891, nor Iijima '611, nor Max '034 teach Applicants' use of lateral constraint and consolidation of the hydrate stream into a monolithic body, adding any or all of the teachings of Ohsol, Satek, or Allen will not produce Applicants' process and result, nor is there anything in the teachings of Spencer, Iijima, or Max to suggest that it would.

Accordingly, because the consolidated, extruded material of Applicants' process is clearly different than the particulate or granular materials produced by processes disclosed in the cited art, and none of the cited art suggests the desirability of forming a consolidated product as a substantially continuous extrusion, it is my opinion that the teachings in Application No. 10/601,234 are novel and nonobvious over the cited art.

11. In conclusion, it is my opinion that the full breadth of the invention as recited in amended claims 1 and 11 is both clear and fully supported by Application No. 10/601,234. Moreover, the recited combination of (1) forming solid particles of gas hydrate as a continuously flowing stream, and (2) consolidating the stream into a substantially monolithic extruded body is not disclosed or suggested in the cited art.

12. I have no financial interest in the above patent application or any related applications, nor am I being paid for my services pertaining to the subject matter of this Declaration.

13. I further state that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with my knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Roddie R. Judkins

Roddie R. Judkins

5/30/2006

Date

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Current Job Duties: Directs all ORNL activities funded by the U. S. DOE Office of Fossil Energy and related Work for Others projects. Program Manager for the DOE Advanced Research Materials Program and coordinates work at six other national laboratories. Director of the ORNL Inorganic Membrane Technology Laboratory. Founder of the Interlaboratory Working Group on Methane Hydrates.

Current Research Areas: Key research areas include:
(1) Microporous inorganic membrane R&D, particularly for separations of interest to DOE Fossil Energy, viz. hydrogen separation, CO2 separation.
(2) Hydrogen production and purification.
(3) Metal-supported solid oxide fuel cell R&D.
(4) Gas-solids, solids-solids, solids-liquid, and liquid-liquid separation materials and devices .
(5) Advanced power systems such as fuel cell-turbine hybrids.
(6) Greenhouse gas emissions separation and capture.

Education: Ph.D. in Physical Chemistry, Georgia Institute of Technology, 1970
M.S. in Chemistry, Tennessee Technological University, 1965
B.S. in Engineering Chemistry, Tennessee Technological University, 1963
Registered Professional Engineer, California

Experience: 28 years of experience at the Oak Ridge National Laboratory in research related to structural and functional materials development.

Publications: Over 150 publications in journals and conference proceedings

Patents: 8 plus 6 pending

Awards: R&D 100 Awards in 1995 and 1999
2000 Federal Laboratory Consortium for Technology Transfer Award for Efforts related to the Iron Aluminide Hot-Gas Filter
1996 Federal Laboratory Consortium for Technology Transfer Award for "Efforts related to the 3M Ceramic Composite Filter
1997 American Museum of Science and Energy Technological Achievement Award for the 3M Ceramic Composite Filter
1998 Elected as Fellow of ASM International
1999 Lockheed Martin Leadership Award

Society Membership: ASME International Gas Turbine Institute, ASM International, and The Society of the Sigma Xi

Recent Publications and Presentations

1. Judkins, RR and BL Bischoff 2004. "Development of Microporous Inorganic Membranes for Separation and Purification of Hydrogen.," 21st Annual International Pittsburgh Coal Conference, Osaka, Japan, Univ. of Pittsburgh/Conf. Advisory Board, 09/13/2004-09/17/2004.
2. Judkins, R.R. and B.L. Bischoff 2004. "Hydrogen Separation Using ORNL's Inorganic Membranes," 2004 Clearwater Coal Conference, Clearwater, FL USA, 04/18/2004 - 04/22/2004.
3. Judkins, R.R., T.R. Armstrong and S.D. Labinov 2004. "The LAJ Cycle: An Innovative and Very High Efficiency Fuel Cell-Turbine Hybrid Power Generation Cycle," 2004 Clearwater Coal Conference, Clearwater, Fl USA, 04/18/2004-04/22/2004.
4. Tortorelli, PF, McKamey, CG, Wright, IG, Pint, BA, and Judkins, RR 2003. "High-Temperature Corrosion of Iron-Aluminide Hot-Gas Filters in a Coal Gasification Environment," Sixth International Symposium on High Temperature Corrosion and Protection of Materials, Les Embiez, France, Les Embiez, France, Electric Power Research Institute (EPRI), Centre National de la Reserche Scientifique, ORNL, and Universite Henri Poincare, 05/16/2004-05/21/2004.
5. Judkins, RR, DE Fain and LK Mansur 2003. "The Potential for Separation and Capture of Carbon Dioxide at High Temperatures Using Inorganic Membranes," 28th International Technical Conference on Coal Utilization & Fuel Systems, Clearwater, FL USA, Coal Technology Association, 04/10/2003 - 04/13/2003.
6. Kad, B, McKamey, C, Wright, I, Sikka, VK, and Judkins, RR, et al. 2002. "Optimization of Oxide Dispersion Strengthened FeCrAl and Fe3Al Alloys: For Advanced Power Systems Applications," Parsons 2003, 6th Intl Charles Parsons Turbine Conference, Dublin, Ireland, 09/16/2003-09/18/2003.
7. Judkins, RR, TR Armstrong and SD Labinov 2002. "LAJ Cycle: A New Combined Cycle Fossil Fuel Power System," ASME TURBO 2002, Amsterdam, Netherlands, ASME, New York, NY USA, 06/03/2002.
8. Judkins, RR, TR Armstrong and SD Labinov 2002. "A Universal Mathematical Model For A New Combined Cycle,Fossil-Fuel Power System (LAJ)Cycle: Part 1 Static Model," ASME TURBO 2002, Amsterdam, Netherlands, ASME, New York, NY USA, 06-03-2002/06-06-2002.
9. Judkins, RR, TR Armstrong and SD Labinov 2002. "A Universal Mathematical Model For A New Combined Cycle, Fossil-Fuel Power System (LAJ) Cycle: Part 2 Dynamic Model," ASME TURBO 2002, Amsterdam, Netherlands, ASME, New York, NY USA, 06/03/2002 - 06/06/2002.
10. Armstrong, TR, Klett, LB, Wilson, DF, Burchell, T, and Judkins, RR 2002. "A Regenerable CO2 Scrubber for Alkaline Fuel Cells," 2002 Fuel Cell Seminar, Palm Springs, CA USA, Courtesy Associates, 11-18-2002 - 11-21-2002.